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Quarterly Technical Report 10 • 31 May 1994

FIELD-EMITTER ARRAYS FOR RF VACUUM MICROELECTRONICS

C.A. Spindt, Program Director
Physical Electronics Laboratory

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SRI Project 2743

Prepared for:

Advanced Research Projects Agency
Defense Sciences Office
Virginia Square Plaza
3701 North Fairfax Drive
Arlington, VA 22203-1714

Attn: Dr. Bertram H. Hui

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13. ABSTRACT *(Maximum 200 words)*

SRI International has completed the tenth quarter of a program to develop field-emitter arrays for vacuum microelectronics. We have met the first-phase program goals of 5 mA total emission, with a current density of 5 A/cm^2 for at least 2 hours and demonstrated modulation of the emission current at a frequency of 1 GHz.

Principal efforts involved the development of a process for forming emitter cones in small-diameter apertures and with improved aspect ratios to reduce stress accumulation on film, modification of reactive ion etching chemistry to eliminate sulfur contamination of the cathode, investigation of galvanic etching resulting from a deionized-water rinse, and methods of fabricating emitters arrays with increased geometric field-enhancement factors and higher packing densities.

Variations in emission test results in cathodes at SRI and in cathodes given high-frequency tests at NRL were investigated, and an emission microscope for use in studying emitter array performance, yield, and reliability was assembled.

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ILLUSTRATIONS

EXECUTIVE SUMMARY

SRI International has completed the tenth quarter of Phase I of a research and development program on the SRI Spindt-type field-emitter-array cathode with a view toward eventual applications in microwave amplifiers. We have met the first-phase goals of 5 mA total emission, with a current density of 5 A/cm^2 for at least 2 hours and demonstrated modulation of the emission current at a frequency of 1 GHz. Our approach has been to identify methods of adapting and modifying the basic cathode structure of microwave operation and to experimentally investigate means of implementing those methods.

During the quarter we have accomplished the following, as documented in detail in this technical report:

- Continued research on basic cathode technology as defined by the goals of the ARPA program and related NRL project (Section 1)
- Developed a process for forming emitter cones in small-diameter apertures and with improved aspect ratios to reduce stress accumulation on film, modified the reactive ion etching procedures to eliminate sulfur contamination due to the recent forced change drom CF_4 to SF_6, investigated galvanic etching resulting from a deionized-water rinse, and researched methods of fabricating emitter arrays with increased geometric field-enhancement factors and higher packing densities (Section 2)
- Investigated the variations in emission test results in cathodes at SRI and in cathodes given high-frequency tests at NRL (Section 3)
- Assembled an emission microscope for use in studying emitter array performance, yield, and reliability (Section 5)
- Planned activities for the period of 1 April through 30 June 1994 (Section 6)

1. INTRODUCTION

SRI International is participating in an effort of the Advanced Research Projects Agency (ARPA) and the Naval Research Laboratory (NRL) to perform research and development on the SRI Spindt-type field-emitter-array cathode with a view toward eventual applications in microwave amplifiers. The current ARPA program is the vehicle for advancing the basic cathode technology for microwave applications (e.g., reducing intrinsic capacitance and driving voltage requirements), and continues the original program plan to establish the characteristics of the cathode in its preprogram state of development, identify methods of adapting and modifying the structure for microwave operation, and experimentally investigate means of implementing those methods. For the NRL program, which began earlier than the ARPA project, SRI has shifted emphasis to the support of NRL's in-house vacuum microelectronics program by providing NRL with state-of-the-art Spindt-type cathodes and consultation on setting up and using cathodes. We have met the first-phase program goals of 5 mA total emission, with a current density of 5 A/cm^2 for at least 2 hours and demonstrated modulation of the emission current at a frequency of 1 GHz.

At the beginning of the program, two areas of development required immediate attention. The first was a materials and processing issue related to providing and maintaining a suitable vacuum environment for the cathodes. The second related to the cathode's inherent high capacitance and means for reducing that capacitance to a level that is consistent with the microwave applications envisioned for the cathode.

Our approach has been to research these two issues in parallel, using an easy-to-build, low-frequency-triode configuration fabricated on a TO-5 header as a test vehicle for materials and processing studies, and at the same time designing and researching fabrication techniques for building high-frequency-cathode structures on dielectric substrates (e.g., quartz or glass). Specific tasks that are being addressed on these related programs are

1. Fabrication of a supply of state-of-the-art cathodes for use in establishing cathode characteristics, and for developing structures, circuits, and procedures for testing the cathodes as triodes

2. Development of a close-spaced anode test configuration that can be used to investigate triode characteristics at low frequency (kHz to MHz) in order to study the known problems with cathode survival under close-spaced anode conditions

3. Development of a circuit for driving the cathodes and demonstrating gain, frequency response, and peak emission levels

4. Studies of advanced cathode structures (geometry, fabrication technology, and processing) for high-frequency operation

5. Investigations (with NRL) of cathode mounting and connecting procedures using practices that are consistent with the microwave goals of the effort

6. Consultations with the NRL staff on the experimental results and applications of the cathode technology

Our recent effort has been directed toward improving high-frequency performance and supplying NRL with cathodes for in-house testing. Progress has been made at SRI on fabrication of smaller cathode geometries for increased transconductance and lower operating voltages, but at NRL it has been difficult to reproduce performance characteristics demonstrated at SRI with cathodes from the same substrates. These difficulties were found to be due in part to previously unsuspected galvanic etching of the emitter tips during SRI's last step of cathode processing, which includes a deionized-water rinse.

2. LOW-CAPACITANCE CATHODE FABRICATION

Our work has been concentrated on film stresses associated with the cone-formation process, verification of the solution to sulfur contamination related to the photoresist, and research on a galvanic etching effect.

2.1 FILM STRESS DURING CONE FORMATION

The previous report discussed problems and solutions associated with high stresses encountered during the cone-formation process. We have now developed processes for forming emitter cones in much smaller diameter gate apertures and with improved aspect ratios (cone height/cone base diameter). These processes also reduce the stress accumulation in the film as it forms, because a thinner closure film is sufficient to close the smaller hole sizes. Experiments with substrate temperature during the cone-formation process have also been helpful in determining that higher substrate temperatures during the process tend to relieve stresses that build up during deposition.

2.2 CONTAMINATION ISSUES

Emission tests with the high-frequency cathode structures have been plagued by contamination problems, as discussed in the previous two quarterly reports. These problems have been associated with a change in the photoresist that made it necessary to modify our reactive ion etching (RIE) chemistry from CF_4 to SF_6 during the molybdenum gate film patterning. A change to SF_6 was necessary because the resist that is now available to us does not hold up to the CF_4 etch well enough. We found by Auger spectroscopy that the SF_6 leaves a surface sulfur residue that is detrimental to the cathode's performance. Similar difficulties have been found by those working in flat-panel display applications of field-emission arrays when sulfur-bearing low-voltage phosphors are used. The solution has been to etch most of the gate

with SF_6 and then switch to CF_4 to finish the process. This procedure appears to have eliminated the sulfur contamination problem.

2.3 GALVANIC ETCHING OF EMITTERS

In previous reports we described unexpected differences in gate aperture diameters from cathode to cathode across a given substrate. These differences have been intermittent, and are still unexplained. Fortunately, the hole patterning has been relatively consistent in recent work. However, during our interaction with NRL we discovered an insidious and devastating effect resulting from our standard processing technique. We have routinely finished our wet-chemistry processing with a half-hour rinse in running deionized water followed by a half-hour bakeout in dry hydrogen. In the many years that we have done this, we have seen no resulting detrimental effect on the molybdenum gate or cones. On the contrary, controlled experiments showed that performance reliability was improved by this procedure as a final cleaning of the cathodes prior their being placed in our vacuum systems for testing.

Since the procedure was designed to be a final and best cleanup prior to testing, we have never done a scanning electron micrograph (SEM) examination of the emitters after the process, because the SEM environment is not UHV clean, and past experience has shown that cathodes exposed to this environment do not perform as well as those that have not been exposed. The relevance of this is that the low-capacitance cathodes have not been inspected in the SEM after the deionized-water rinse that is done just prior to our placing the cathodes in storage/shipping vacuum vessels or into a vacuum chamber for testing.

We recently received SEMs of cathodes that had been tested at NRL, and we were astonished to see vast differences in the emitter tips in the NRL-tested cathodes as compared to cathodes from the same batch that were tested at SRI. Figure 1 shows this effect very clearly. Figure 1a is a micrograph made at SRI of a cathode on substrate 100L-E11-22 after cone formation and before the gate patterning, dicing, and final cleanup for test. Figure 1b is a SEM made at NRL of cathode 100L-E11-22K from that substrate after gate patterning, dicing, cleaning, hydrogen firing, shipping to NRL, and testing at NRL. Clearly, there has been a change in the cones.

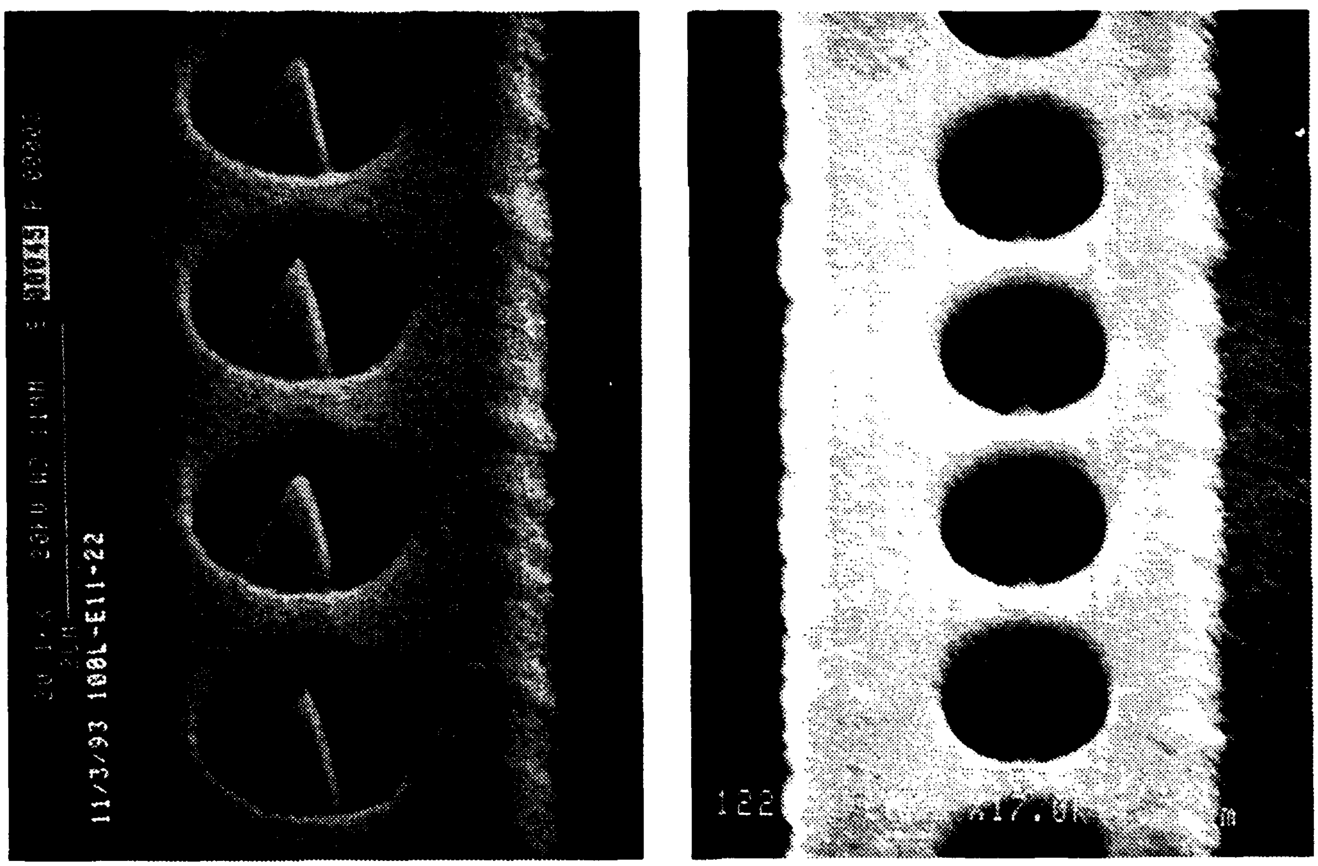
A step-by-step review of our process showed that the molybdenum electrodes on the low-capacitance cathodes are etched slowly during the deionized-water rinse. This appears to be the result of galvanic etching in the water. Gold contact pads have been added to the high-frequency cathode structures for bonding to microstrip lines, and the presence of the gold on the substrate apparently causes a slow galvanic dissolution of the molybdenum cones and gate in the deionized water. This etch is also rather remarkable in that the cones remain sharp, but are reduced in size as though they "shrank."

As a confirmation of the assumption that we were dealing with a galvanic etching effect, a test cathode was monitored at each step of the process after cone formation. No change in the cone shape was detected until after a half-hour deionized-water rinse. Figure 2 illustrates the change. Figure 2a shows the cones before the deionized-water rinse, and Figure 2b shows the same cones viewed at the same angle and magnification after the half-hour deionized-water rinse. Etching has clearly taken place. The Figure 2 SEMs are substandard because of the gate film's being patterned prior to the microscopy, so that bare glass on the substrate becomes charged in

increasing the emitter tip packing density. The second is a study of ways of making high-aspect-ratio (cone height to base diameter), sharp cones for use with the smaller gate apertures so that it may be possible to fabricate smaller apertures and larger packing densities while maintaining the oxide thickness and capacitance. This is important, because if the capacitance is increased in the same proportion as the transconductance, the net gain in cutoff frequency is zero.

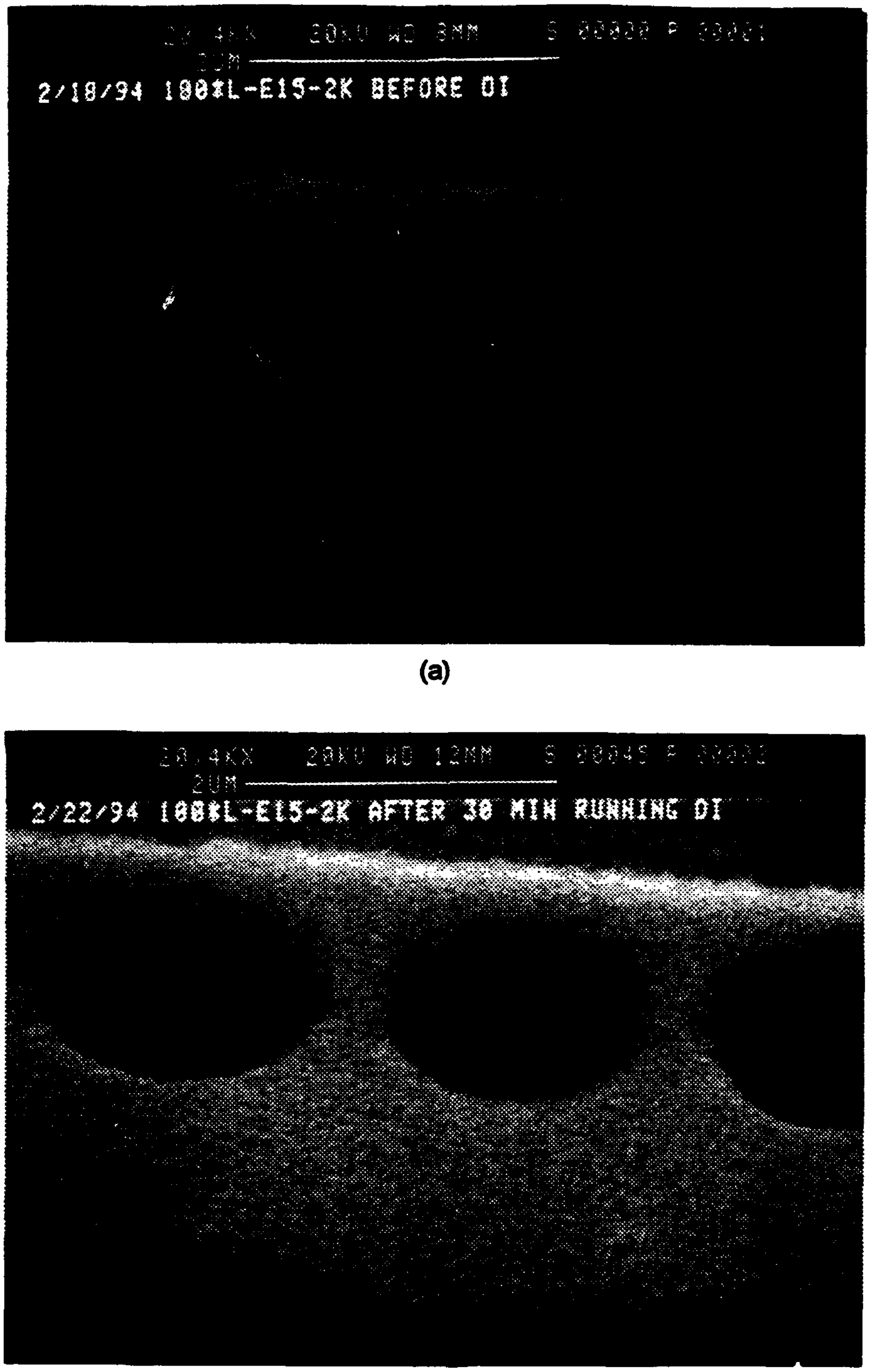
Submicrometer-hole diameters have been formed in standard SRI-supplied silicon-based test samples by Hughes Research Laboratories (HRL) using an experimental focused ion beam lithography system under development at HRL. Patterns of 100 holes in a 10 x 10 array have been lithographed in poly(methyl methacrylate) with hole diameters of approximately 1000 Å and center-to-center spacing of both 1 μm and 0.5 μm. The samples are then returned to SRI for etching and cone formation.

The original patterns were made in cathode substrates with a 2500-Å thick oxide layer, because this work was started at the same time as investigations into means for fabricating cones with higher aspect ratios, and thus only relatively short (~1:1 aspect ratio) cones were originally available.



(a) (b)

Figure 1. Scanning electron micrographs of cones from substrate 100L-E11-22: (a) at SRI as fabricated, (b) at NRL



(a)

(b)

Figure 2. Scanning electron micrograph of three emitter cones in a cathode array: (2) as fabricated, (b) viewed at the same angle and magnification after a half-hour rinse in deionized water

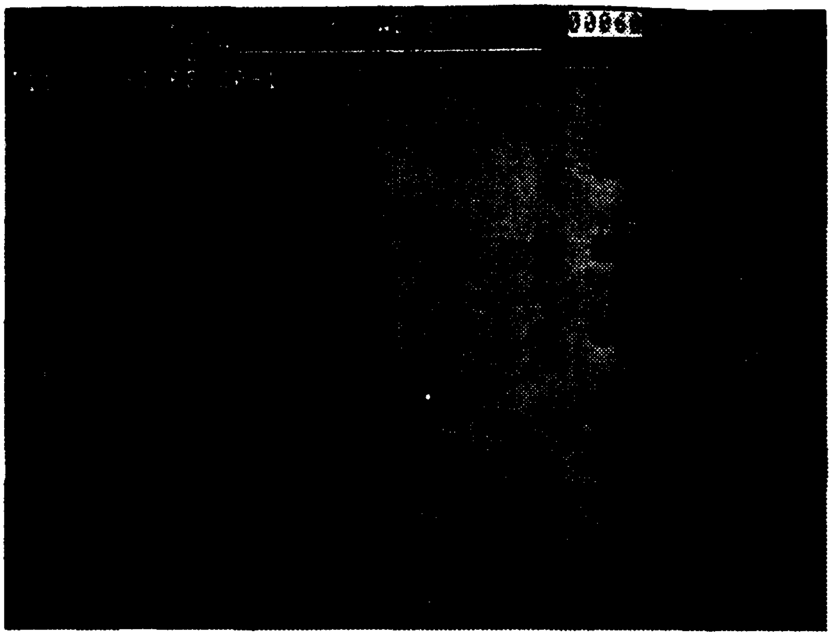
The first patterns were inadequate because of uniformity issues associated with the fact that the resist used was too thin to withstand the required etching processes. However, a double resist procedure was then used to successfully pattern holes about 0.25 μm in diameter in a 10 x 10 array with 1-μm spacing. Cones were then formed in the holes using our standard cone-forming process. Figure 3a is a SEM of one of the first arrays made in this way, and Figure 3b is a SEM of a portion of a low-capacitance array of cones on 2-μm centers fabricated with photolithography. Until now, the low-capacitance structure has been our standard size. The cones in the submicrometer array appear to be short and blunt, but this is mainly an artifact of the scale. The holes are so much smaller than we are used to looking at that the tips seem big by comparison, but close inspection shows that there is not much difference between the tip radii in Figures 3a and 3b. Emission tests will be made with the small arrays during the next reporting period. We are also planning to begin fabrication of small-hole arrays in low-capacitance structures.

3. EMISSION TESTS

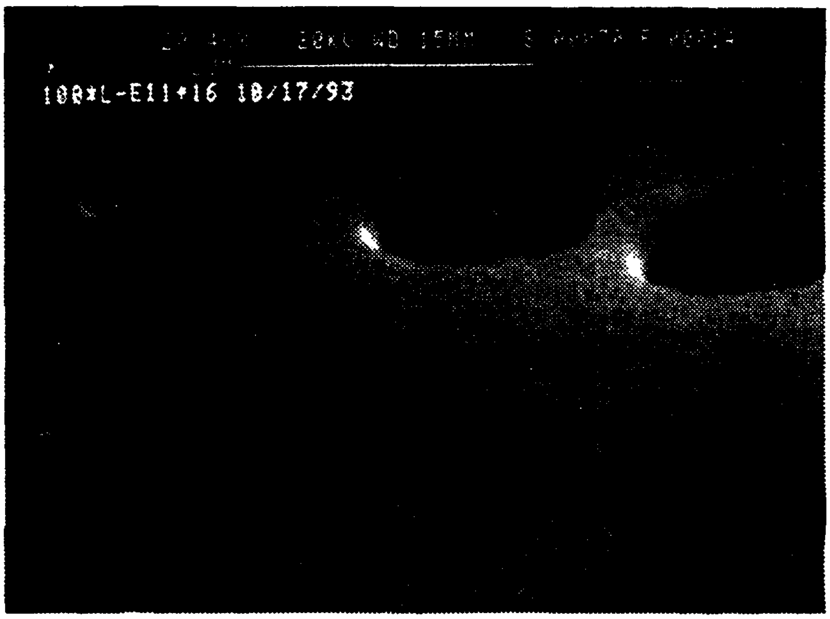
In-house testing of high-frequency cathode samples at NRL has met with limited success, as discussed in our previous quarterly report. Emission has been insufficient to permit high-frequency experiments. Some cathodes may have suffered mechanical damage during shipping, while others were very different from what we had seen during SEM examinations at SRI.

We now know that the cathodes were so different because of the galvanic etching action in the deionized-water rinse. As soon as we determined this etching effect, we processed three low-capacitance cathodes from each of four different substrates (24 cathodes per substrate) using alcohol instead of deionized water for the final rinse step. All twelve cathodes were then mounted in TO-5 headers and placed in our standard UHV-vacuum ion-pumped test system. The standard procedure is to rough the system with an oil-free turbomolecular pump, switch to an ion pump with a titanium sublimator at about 10^{-6} torr, and bake at 750 K for 48 hours. At the end of that time the pressure is usually about 1×10^{-9} torr, and the cathodes are turned on after the chamber is cooled to room temperature.

One of the twelve cathodes in this test had an electrical short between the base and gate electrode at turn-on, but the other eleven all produced 1 mA of emission without any difficulty. Figures 4, 5, 6, and 7 show current vs. voltage data for the eleven cathodes that worked well. The spread in the data within each group occurs because some cathodes have 250 tips on 5-μm centers, and others have 625 tips on 2-μm centers. Variations are also due in part to small differences in ß factors (gate aperture diameters and associated tip sharpness) between individual cathodes and the limits of the uniformity it is possible to achieve with the apparatus presently available for fabrication processes.



(a)



(b)

Figure 3. Scanning electron micrographs showing cones formed in holes patterned with double resist procedure (a) 10 x 10 array with 0.25-μm-diameter gate apertures, (b) portion of a "standard" low-capacitance linear-cathode array of cones on 2-μm centers

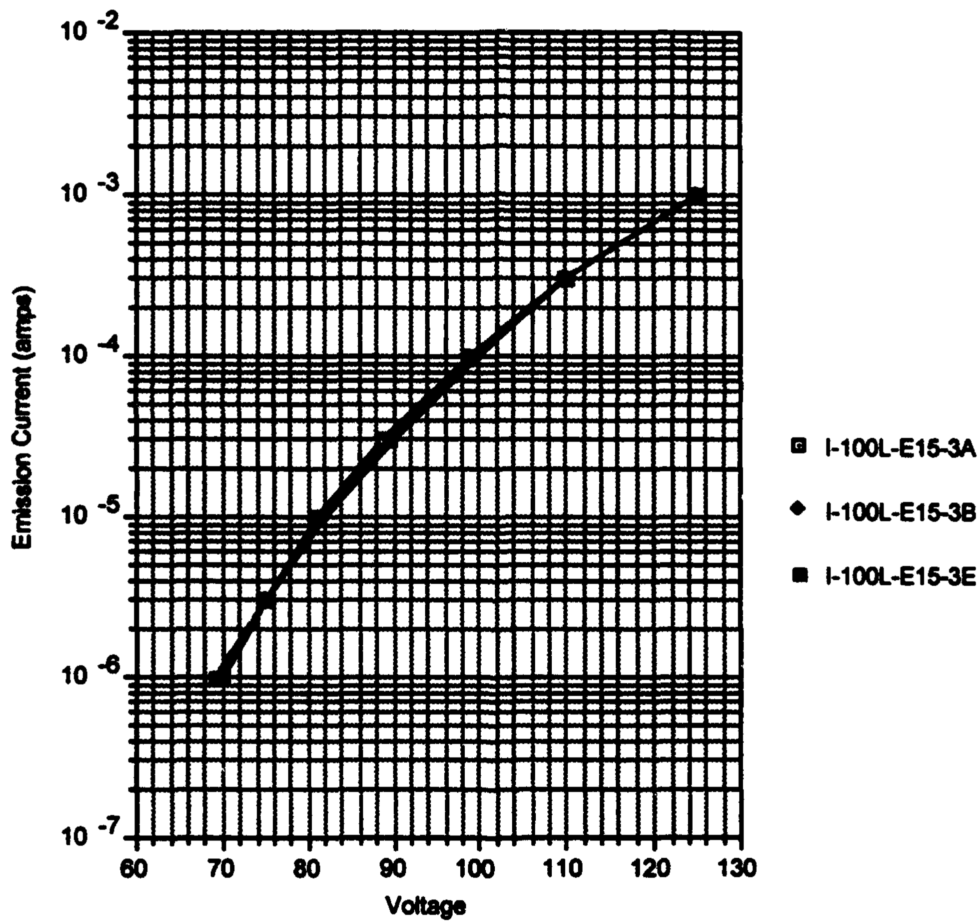


Figure 4. Current/voltage curves for cathodes 100L-E15-3A, B, and E

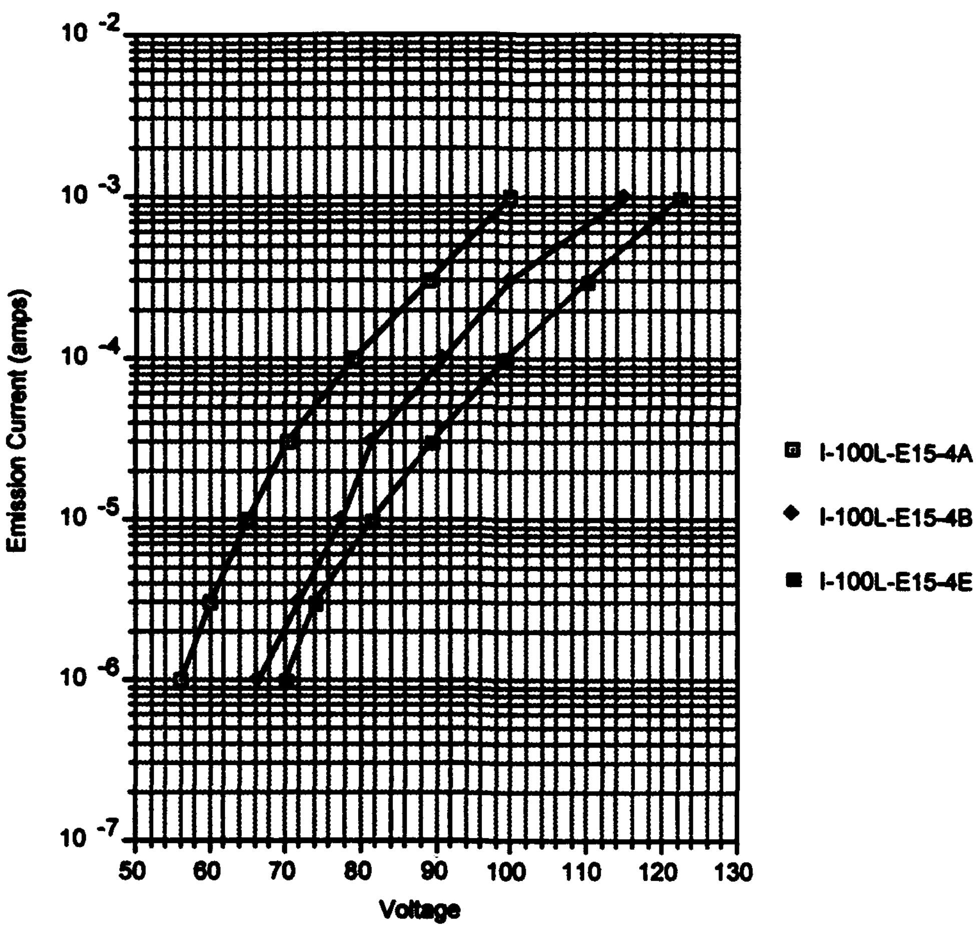


Figure 5. Current/voltage curves for cathodes 100L-E15-4A, B, and E

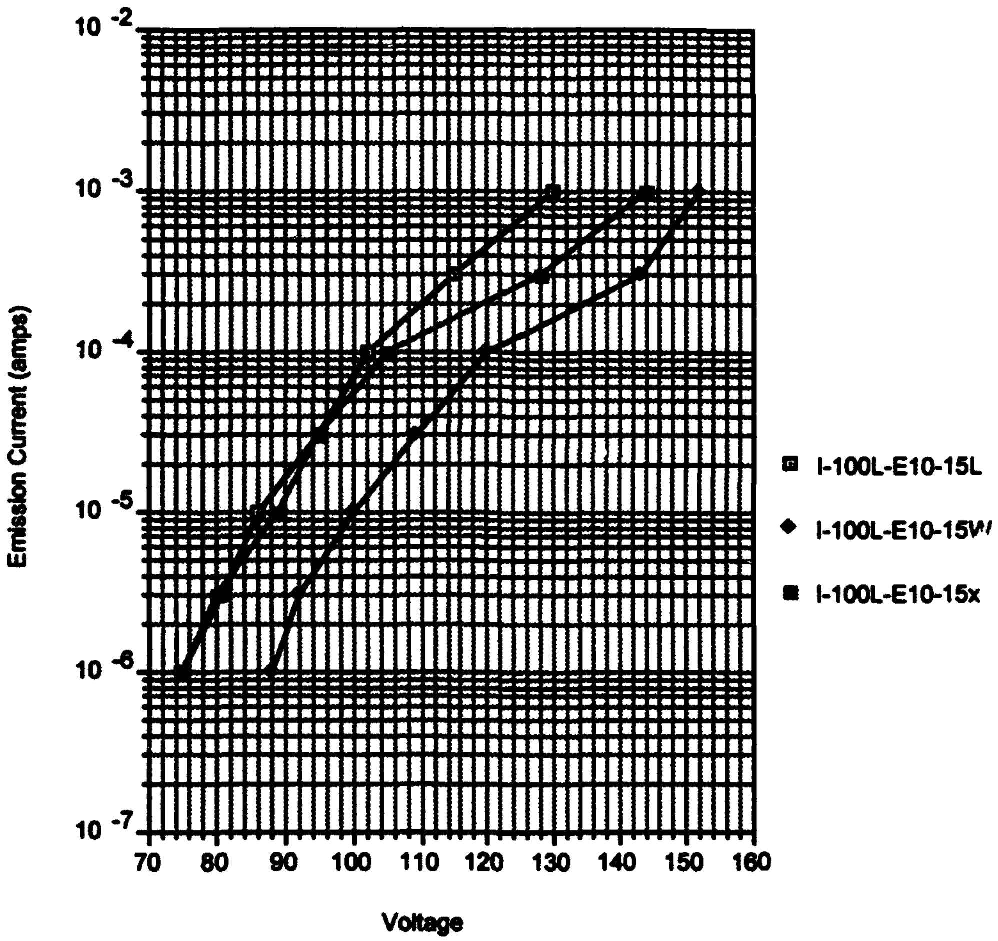


Figure 6. Current/voltage curves for cathodes 100L-E10-15L, W, and X

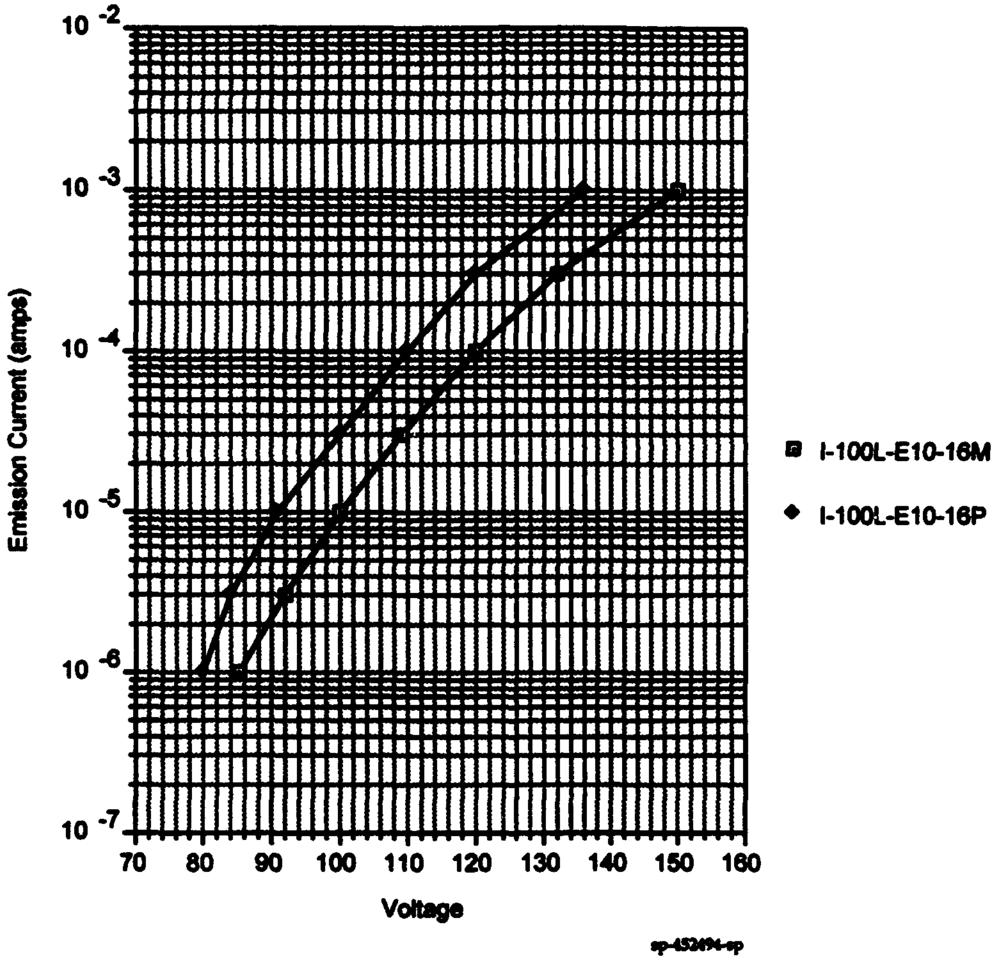


Figure 7. Current/voltage curves for cathodes 100L-E10-16M and P

Untested cathodes from these same four groups were sent to NRL for in-house testing. The cathodes tested at SRI cannot be retested at NRL because it is impossible to lead bond to the gold contact pads after the cathodes have been through a test. We assume that chrome diffusing through the gold during bakeout forms an oxide on the surface when it is exposed to air. However, it has been agreed that NRL will supply SRI with one of it high-frequency mounts so that SRI can mount, *pretest*, and ship a high-frequency cathode to NRL.

4. HIGH-FREQUENCY MEASUREMENTS

No high-frequency measurements were made during this period.

5. PROCESSING TO IMPROVE PERFORMANCE, YIELD, AND RELIABILITY

Work is continuing on our study of the emitter array's performance and its improvement. We have conducted Müller microscope imaging, Auger spectroscopy, and observations of the effects of plasma treatments and various gas environments with the aid of the Müller microscope. We directed our efforts toward assembling and activating an emission microscope obtained on loan from Hughes Aircraft in Torrance, California. The instrument is elegantly simple in principle, but does require some learning and alignment to achieve a useful image. The objective is to image an array with sufficient magnification and resolution to resolve each individual tip so that we can determine uniformity of emission from tip to tip over the array, as well as the effects of various treatments on the uniformity of emission. At the end of the reporting period, the microscope was ready for its first trial.

6. WORK PLANNED

We will continue with cathode fabrication and emission measurements so that we can evaluate the effect of processing steps designed to improve yield and reliability and to maintain a supply of cathodes for NRL's in-house testing.

Work with the emission microscope will continue, as will fabrication processes to produce the smallest possible gate aperture diameters and tallest, sharpest cones in the holes. We will perform emission tests with the small gate-aperture structures.